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(abstract)

The study of the principles of burning of ammonium perchlorate is of great interest owing to the anomalies which have been ~~xxxx~~ revealed by studies of the process. ~~xxxx~~ One of the author's experimentation showed the burning of perchlorate to be very sensitive to changes in experiment conditions, and in particular to the jacket -unjacketed specimens 7 mm in diameter at pressures exceeding 150 atm, in contrast to specimens with a 4 mm diameter. The dependence of the burning velocity on pressure has an unusual form: an increase in pressure above 150 atm entails a drop of burning velocity. Temperature profiles were obtained in the 40-350 atm pressure range. Anomalous dependences of surface temperature and heat release in the condensed phase of pressure were established.

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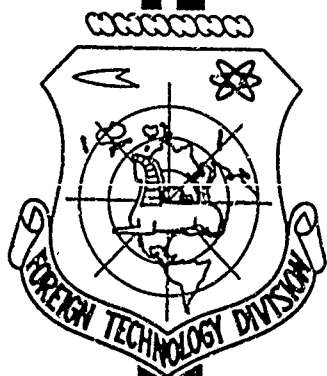
TRANSLATION

TEMPERATURE PROFILES UPON BURNING OF AMMONIUM PERCHLORATE

By

V. K. Bobolev, A. P. Glazkova et al.

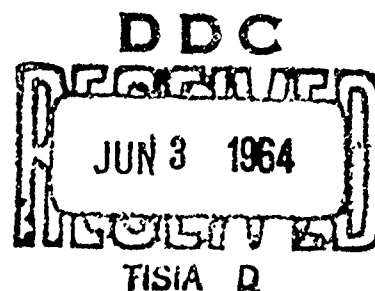
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TEMPERATURE PROFILES UPON BURNING OF AMMONIUM PERCHLORATE

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The study of the principles of burning of ammonium perchlorate is of great interest owing to the anomalies which have been revealed by studies of this process. Friedman, Nugent et al. [1] discovered the phenomenon of the higher and lower limit of burning with respect to pressure, and with the help of thermocouples (junction $\sim 50 \mu$) also measured the maximum temperature of the flame, which is 930° and increases slightly when the pressure is increased to 150 atm. Later, Levy and Friedman [2], while continuing the study of perchlorate burning, eliminated the phenomenon of the higher limit by using an asbestos jacket.

However, as shown by the experiments of one of the authors of this article [3], the burning of perchlorate is very sensitive to changes in experiment conditions, and in particular of the jacket; with unjacketed specimens 7 mm in diameter (at pressures exceeding 150 atm), in contrast to the specimens with a 4 mm diameter which were used by Friedman, burning occurs, but unsteadily with pulsations, and the dependence of the burning velocity on pressure has an unusual form: an increase in pressure above 150 atm entails a drop of burning

velocity*.

A drop in the burning velocity with increased pressure was also observed by A. F. Belyayeva and A. I. Korotkova in experiments with potassium picrate.

This phenomenon is unusual. A proposed explanation of this fact has been expressed by K. K. Andreyev and Song Quan Cai [5] who have linked it with phase conversion, inherent to perchlorate at 240° , proposing that at a certain pressure the surface of burning perchlorate must reach this temperature (whereupon at this temperature there is a conversion from an orthorhombic to a cubic variation which, according to the data of Bircumshaw [6] disintegrates at a much lower rate).

In light of the aforementioned, measurement of temperature distribution in the condensed and gas phases during ammonium perchlorate burning is important, because the temperature measurements can shed light on its mechanism. Arden, Poling and Smith [7] attempted to determine the surface temperature of burning perchlorate with the help of an IR (infrared) pyrometer and the maximum temperature by methods which have been cited above [1]. They established that at atmospheric pressure $T = 450 \pm 30^{\circ}$ and is independent of burning velocity or the nature of fuel admixture; they also assumed that the leading mechanism during burning is sublimation.

We studied the temperature distribution during the burning of ammonium perchlorate using the method of fine thermocouples, which was developed by A. A. Zenin [8].

For measurements we used II-type thermocouples W + Re to W + Re (5 and 20% Re), circular, with a diameter of 15 and 30 μ , and laminar, with a thickness of 3.5 and 7 μ , respectively.

* When the pressure was increased we observed a drop in the burning velocity for the 12 mm specimens, which under these conditions is considerably greater than the critical diameter of burning (see [3]).

The experiments were set up with 7 mm specimens of unsifted ammonium perchlorate (compressed to about $1.93\text{--}1.94\text{ g/cm}^3$), for which the drop in burning velocity with pressure was sharpest. We conducted experiments in an atmosphere of nitrogen with constant pressure in the 30-350 atm range. During these experiments the velocity and character of burning were simultaneously recorded on film with the aid of a photorecorder and the temperature profile was recorded on an N-700 oscillograph.

It was revealed, in particular, that when the pressure is higher than 150 atm together with the shutdown of burning, regular oscillations of temperature occur in the gas phase with an interval of $\sim 50\text{ }\mu\text{ sec}$ and an amplitude of oscillation to 500° ($1000\text{--}500^\circ$). At the same time it should be emphasized that the fluctuations in the temperature of the flame and the plateau on the temperature recording are in good agreement with the reduction of the luminous profile and the shutdowns on the photographs of burning. This gave us the basis for calling the 160-350 atm range of pressures the range of unstable burning.

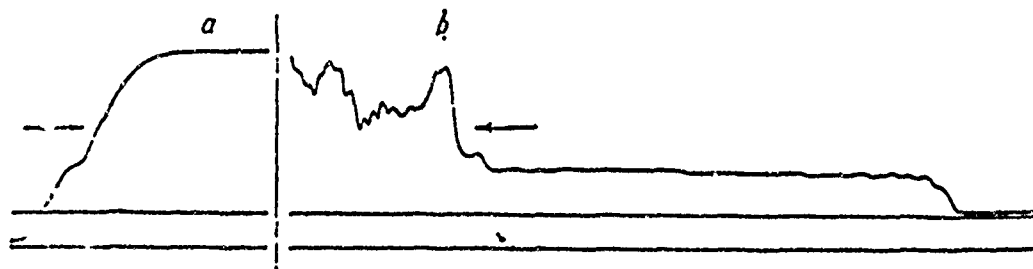


Fig. 1. Typical oscillograms of burning. a) in the stable range, $P = 50\text{ atm}$; b) in the unstable range, $P = 250\text{ atm}$.

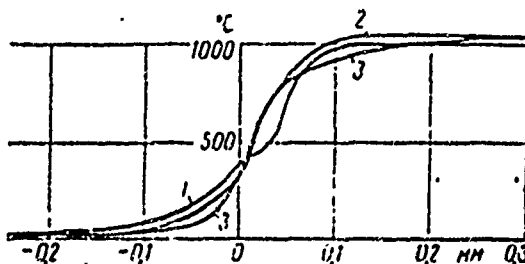


Fig. 2. Temperature profiles when $P = 50$ (1), 100 (2), 150 (3) atm.

Figure 1 shows the most typical oscillograms for stable and unstable burning, and Fig. 2 shows the temperature profiles derived by analysis of the oscillograms in the stable range. Oscillograms in the range of unstable burning (160-350 atm)* are characterized by the presence on most of the oscillograms of a plateau with a stable temperature of about 270° and temperature pulses in the flame. We note that in certain experiments burning dies out after having reached the thermocoupler, in these cases the surface of the burned out charge was perfectly horizontal, and the maximum recorded temperature was $\sim 270^{\circ}$, which was taken as the surface temperature.

In the range of stable burning the surface temperature was determined by Zenin's method [8] with respect to the maximum intensity of heat release in the condensed phase during analysis of the oscillograms, and also by direct measurements with loaded thermocouples conforming to the methods proposed by P. F. Pokhil [9].

Figure 2 shows total heat release, heat release in the condensed and gas phases, and also heat arrival from the gas phase (q) as functions of pressure.

In the 40-150 atm range of pressures (stable regime) we observed an increase of the total heat release (1), which has been linked to its strong increment in the gas phase (2), because the heat release in the condensed phase decreases with an increase of pressure. However it would be incorrect to consider that an increase of the burning velocity is only associated with the heat release in the gas phase, because the heat supply from the gas phase was small (4) and it changed

* At present there is no possibility of a final analysis of oscillograms of unstable combustion since photo-recording of the velocity does not permit an accurate calculation of its value at sections of retardation and the time of burning shutdown. The oscillograms will be analyzed after additional experimentation.

slightly with the pressure. Heat release in the condensed phase was an order higher, but it decreased sharply with an increase of pressure and therefore we were not able to explain the observed increase in burning velocity by the usual heat mechanism. The explanation of this phenomenon must probably be sought in the chemical nature of those reactions, the mechanism of which can change with the pressure. Theoretically a sharp increase of dispersion is also possible, however in our experiments it was greater the lower the burning velocity (at 50, 250-350 atm)*.

A supposition was mentioned above [7] concerning the decisive role of the sublimation of solid perchlorate in the process of burning, but when burning ammonium a considerable amount of heat is released in the condensed phase (80-120 cal/g) (Fig. 3), whereas sublimation should have been accompanied by its absorption. Furthermore, upon sublimation the surface temperature should have increased with the pressure, but not conversely, as derived from our experiments. It is evident from Fig. 4 that the surface temperature of burning perchlorate decreases with an increase of pressure and in the range of unstable burning is approximately the temperature of phase transition. We noted in passing, that on certain oscillograms a small discontinuity having an essential thermoneutral effect was observed in the range of temperatures of the order of 250°, which is possibly associated with the temperature of phase transition (Figs. 4, 2).

It was noted above (see Fig. 3), that heat release in the condensed phase decreases with an increase in pressure, which was not previously observed for any of the materials studied. At 150 atm heat

* In connection with this observation, P. F. Pokhil noted that, evidently, the mechanism of ballistics-powder burning, i.e., burning with the participation of dispersion and the formation of a fume-gas phase, may be applied to the burning of ammonium perchlorate.

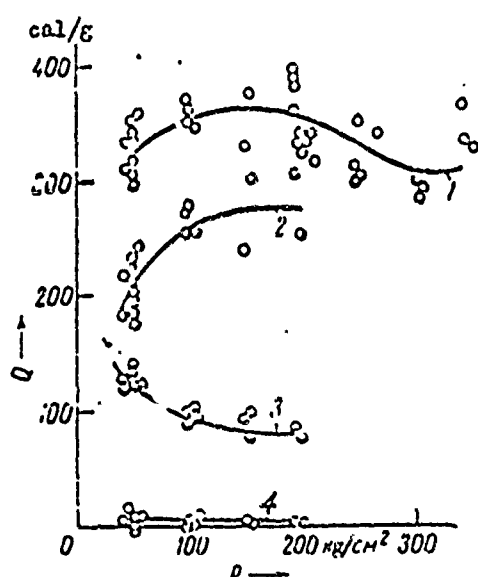


Fig. 3. Dependence of total heat release (1), of heat release in the gas (2) and condensed (3) phases, and also of heat influx from the gas phase (4) on pressure when burning ammonium perchlorate.

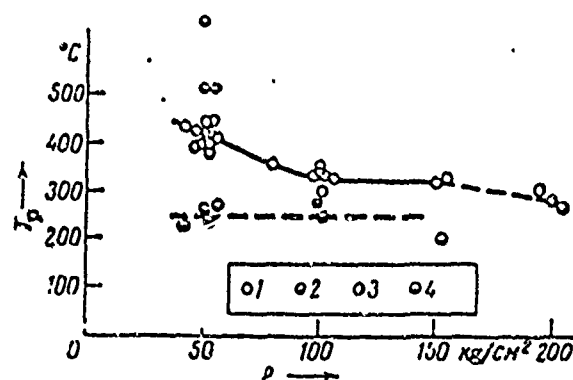


Fig. 4. The dependence of temperature at the burning surface of ammonium perchlorate on pressure. 1) T_p , determined by a spike on the oscillogram; 2) T_p , determined with a loaded thermocouple; 3) T_p , the same, at 200 atm, with stable burning of preheated (to 40-50°) specimens; 4) temperature discontinuity in the distribution of temperatures in the condensed phase during certain experiments of ammonium perchlorate burning.

release amounts to 80 cal/g. During experiments in the range of unstable burning we observed the dying out of the specimens having a surface temperature $\sim 270^\circ$. If we calculate the amount of heat necessary for raising the condensed phase to this temperature, it will also equal 80 cal/g. Thus it was assumed that this amount of heat is critical and is the threshold which separates the range of stable burning from that of unstable burning. If the condensed phase receives more than 80 cal/g, burning is steady, becomes pulsating on the boundary, and with a lesser quantity of heat dies out. Heat release in the condensed phase is determined by the surface temperature which, due to a mechanism as yet unknown to us, decreases with pressure (see Fig. 4), and as the temperature of phase transition is approached the heat release in the condensed phase should decrease still more sharply,

because phase transition occurs with an absorption of heat. This may be the reason for the reduction of velocity and in individual adverse cases — the reason for the shutdown of burning.

The results, shown in Fig. 4 in the 50-150 atm range, are striking not only for the drop of surface temperature with an increase of pressure, i.e., with an increase of burning velocity, but also for the lower value of temperature. Known data concerning kinetics of perchlorate decomposition, which have been obtained at temperatures to 280° [5], and which have also been extrapolated to 420°, yields a velocity of perchlorate gasification several orders less than the measured rate of decrease in the amount of perchlorate during burning. Apparently, the kinetics and mechanism of gasification at a relatively low temperature in experiments dealing with perchlorate decomposition are different from the kinetics and mechanism of perchlorate gasification which takes place at the same or a much higher temperature at the surface of burning perchlorate. One of the authors proposes the possibility that during burning there is catalysis of perchlorate decomposition at the surface by reaction products and active centers passing from the flame zone to the surface of the condensed phase. The fact is that the presence of the heat flow from the flame zone to the surface automatically indicates a diffusion of molecules (including the radicals) from the flame zone to the surface. These active reaction products can render an effective catalytic reaction.

This supposition relates, naturally, not only to the burning of perchlorate, but also to the burning of any condensed system in which there is a heat flow from the reaction zone in the gas phase to the surface of the condensed phase, heat conductivity in gas being accomplished by diffusion of "hot" (including active) molecules. This supposition also obviously includes a possible mechanism, by which the

gas phase controls the decomposition of the condensed phase. This is particularly essential in those cases, when heat transition from the gas phase is not enough, and heat release in the condensed phase is great and almost ensures maintenance of the specified surface temperature.

Maximum burning temperature increases first with an increase in pressure, passing through a maximum value of 1060° at 100-150 atm, and then begins to drop even though the pressure is increased.

As a result of work, temperature profiles were obtained in the 40-350 atm pressure range, which indicate two regimes of ammonium perchlorate burning: stable (40-150 atm) and unstable (160-350 atm).

Anomalous dependences of surface temperature and heat release in the condensed phase on pressure were established — they fall when the pressure is increased.

These assumptions concerning the reasons for the drop in burning velocity with an increase in pressure and concerning the catalytic effect of products diffusing from the reaction zone in the gas phase to the surface indicate a gasification reaction of the condensed phase.

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